

...the degree of chlorination in all cases increased with a rise in temperature (25-61% Cl by weight); and showed also that the aromaticity of the sample chosen was 42%. The results of exhaustive chlorination give an indication of the percentage aromaticity because the process is accompanied by isolation of the peripheral chains of the aromatic part of the molecules. Chlorination was found to give more reliable data on aromaticity than oxidation.

C.A.

KRETOV, A.S.

✓ Acylation of aromatic amines with maleic anhydride.
A. S. Kretov and N. P. Kulchinskaya (P. N. Gerasimov
Chem. Technol. Inst., Dnepropetrovsk), *Zhur. Obshch. Khim.* 25, 2474-9 (1955).—The curves of the acylation rates of various aromatic amines with maleic anhydride

(I) at 10° and 30° in equimolar mixts. are given. The values of log k (in l./mole-min.) are as follows: PhNH₂, 0.46; o-MeOC₆H₄NH₂, 0.3; p-MeC₆H₄NH₂, 0.21; 2-C₆H₄NH₂, 0.14; p-ClC₆H₄NH₂, -0.2; o-PhN₂C₆H₄NH₂, -0.3; o-ClC₆H₄NH₂, -1.25; m-O₂NC₆H₄NH₂, -1.64. This series parallels the basicity of the amines. Stirring the amines with 10% excess I in C₆H₆, PhCl, or Me₂CO thus gave the following ArNHCOCH₂CHCO₂H (Ar shown): 97% Ph, m. 193-4°; 93% p-PhN₂C₆H₄, m. 208-9°; 93% m-Me-C₆H₄, m. 167°; 96% o-O₂NC₆H₄, m. 134°; 96% m-HO-C₆H₄, m. 185°; 93% 2,5-Cl₂C₆H₃, m. 143°; 95% 2,4,5-Cl₃-C₆H₂, m. 162°; 94% 4,3-Cl₂(O,N)C₆H₃, m. 109-10°; 92% 4,5,2-Me(O,N)(MeO)C₆H₃, m. 135-6°; 94% 4,2-Et(O,N)C₆H₃, m. 137-8°; 92% 4,3-Me(O,N)C₆H₃, m. 126°; 95% 2,5-Me(O,N)C₆H₃, m. 154°; 93% 4,2-ClMeC₆H₃, m. 129-30°; 95% p-AcNHCC₆H₄, decomp. 218°. G. M. Kosolapoff

SHENBOR, M.I.; KRETOV, A.Ye.; SAVIN, M.I.

Effect of organic solvents on chlorinated lignite. Ukr.khim.
zhur. 22 no.4:546-549 '56. (MIRA 10:10)

1.Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Solvents) (Lignite)

KRE TOV, A.F.

Chem 2
1 Acrylamides, their preparation, and properties.
A. E. Kretev and M. E. Kulchitskaya, Izv. Akad. Nauk SSSR, Khim. (1958)
Chem. Abstr. 52, 135-13; J. Gen. Chem. U.S.S.R. 26, 231-2 (1958)
(Engl. translation).--PAAHCOOH, CH₃COOH (1.0 g) at
100-30° was treated with 3 g. P₂O₅ (temp. rose in several
minutes); the cooled melt was washed with H₂O and extd.
with CCl₄, yielding 64% N-phenylamide (II), m. 58-6°;
thus were prepd: N-p-tolyl analog, 51%, m. 141-6°; and
N-p-anisyl analog, 51%, m. 115°. I 195 g. in xylene
treated at reflux with 1.7 ml. PCl₅ and boiled 3 min., cooled,
filtered and coapd. gave II; if the refluxing continues 0.75
hr. there is formed chlorosuccinimide, m. 115-19°. I
warmed with PCl₅ (9.0 g. I and 11.5 g. PCl₅) gave an exothermic
reaction and on cooling yielded N-phenylamide of
chlorosuccinic acid, 67%, m. 115-19°; SOCl₂ reacted similarly.
Thus were prepd. the following N-acylamides of
chlorosuccinic acid: N-p-tolyl, 70%, m. 157°; N-m-tolyl,
70%, m. 137-8°; N-o-tolyl, 70%, m. 120-1°; N-ethoxy-
phenyl, 66%, m. 125°; N-p-anisyl, 71%, m. 112-8°;
N-m-nitrophenyl, 79%, m. 145-6°; N-p-nitrophenyl, 94%,
m. 173-4°; the use of SOCl₂ is best made at 12-15°; the
reaction running 1-2 hrs. Refluxing I with 4 molar Ac₂O
1.5 hrs. and dilg. with H₂O gave AcH₂CH₂CH₂CH₂OH, 74%.
Similarly were prepd: 75% m-H₂C₆H₄NH₂Ac; 70% p-isomer;
70% o-ClC₆H₄NH₂Ac; 70% p-isomer. Heating 0.03 mole I
with 1 g. NaOAc and 0.03 mole Ac₂O 20-30 min. at 40-60°
gave II. Thus were obtained the following N-acylamides. 1/2

Kretov, A. E. and Golchitskaya, N. E.

amides (aryl group shown): o-tolyl, 78%, m. 115-20°; p-chlorophenyl, 80%, m. 63-40°; p-chlorophenyl, 70%, m. 100-10°; 2-naphthyl, 92%, m. 154-5°; p-benzocyclophenyl, 88%, m. 150-1°; 2,6-dichlorophenyl, 64%, m. 100-10°; 3,4,5-trichlorophenyl, 62%, m. 104°; 4-chloro-3-nitrophenyl, 60%, m. 180°; 4-nitro-3-chlorophenyl, 87%, m. 61°; 3-nitro-4-tolyl, 60%, m. 103°; 3-nitro-2-tolyl, 67%, m. 107-8°; 4-chloro-3-tolyl, 91%, m. 68°; 4-acetamidophenyl, 81%, m. 168°. The ease of cyclization of I indicates the cis structure for it and its analogs.

G. M. Kozlov

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73-3-10/24

AUTHOR: Kretob, A. Ye.

TITLE: Acylation of Arylsulphamides and N,N-Dichloroaryl Sulphamides by Fatty Acid Anhydrides. (Atsilirovaniye Arilsul'famidov i N,N-Dikhlorarilsul'famidov Angidridami Kislot Zhirnogo Ryada)

PERIODICAL: Ukrainskiy Khimicheskii Zhurnal, 1957, Vol. 23, No.3, pp. 344-347 (USSR).

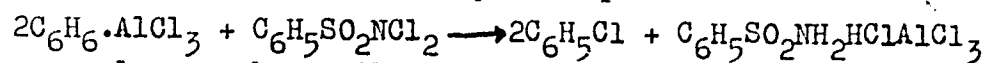
ABSTRACT: Acylation of arylsulphamides by acetic anhydride is considerably accelerated in the presence of sulphuric and other acids (Refs.: 1 and 2). A 70-80% yield of acyl derivatives of benzene-, toluene- and p-chlorotoluol-sulphamides was obtained when H_2SO_4 was present during the reaction. The catalytic effect of aluminium chloride on the acylation of arylsulphamides and chloramine-B was investigated. The acylation of arylsulphamides in the presence of anhydrous aluminium chloride in a solution of aromatic hydrocarbons was studied for toluene-, benzene-, β -naphthalene- and p-chlorobenzene-sulphamides. In the first stage the complex $ArSO_2NH_2 \cdot AlCl_3$ is formed which is soluble when heated in excess benzene or chlorobenzene. This complex reacts with the acid anhydrides. This reaction can be confirmed by analysing the obtained salt

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73-3-10/24

Acylation of Arylsulphamides and N,N-Dichloroaryl Sulphamides by Fatty Acid Anhydrides.

and by the separation of 2 molecules of HCl. Although the initial stages are more complex in the case of N,N-dichloroaryl sulphamides, yet the same end-products are obtained. The first stage of the reaction is carried out in a solution which is able to chlorinate the aromatic hydrocarbon. At this stage equimolecular quantities of anhydrous aluminium chloride and N,N-dichloroarylsulphamide react during cooling and agitation. On analysing the complex after distilling of the solvent the first stage of the reaction can be represented by the equation:



The complex can be easily acylated in a benzene solution by an equimolecular quantity of acetic anhydride. HCl is separated even at 45 - 50°C, the separation is most effective at 60 - 70°C and is terminated 3 - 4 hours after the first separation of 1 mole of HCl. Table I gives the composition of the mixture, the duration of the reaction and the yield of HCl and acetylarylsulphamide during the acylation of arylsulphamides. The preparation of the

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73-3-10/24

Acylation of Arylsulphamides and N,N-Dichloroaryl Sulphamides by Fatty Acid Anhydrides.

complex, of the mixed acetyltoluolsulphamide are described. The melting point and nitrogen content in the synthesised N-acetylarylsulphamides are given in Table 2. N,N-dichlorobenzenesulphamide and arylsulphamides form, in the presence of aluminium chloride, with fatty acid anhydrides the complex aluminium salts of N-acetylarylsulphamides with acetic and hydrochloric acid. After decomposition with water the N-acetylarylsulphamide is formed. A 70 - 85% yield of acyl derivatives is obtained when acylation is carried out in the presence of aluminium chloride. The hydrolysis, alcoholysis and acidolysis of the complex salts were investigated. There are 2 tables and 3 references, 2 of which are Slavic.

SUBMITTED: January, 16, 1957.

ASSOCIATION: Dnepropetrovsk Chemical Technology Institute.
(Dnepropetrovskiy Khimiko-Tekhnologicheskii Institut)

AVAILABLE: Library of Congress.

Card 3/3

KRETOV, A. Ye.
KRETOV, A.Ye.; SHEVCHENKO, V.I.

Acylation of N,N-dichlorarylsulfamides and arylsulfamides by
anhydrides of aromatic acids. Ukr.khim.zhur. 23 no.4:493-495
'57. (MIRA 10:10)

1.Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Acylation) (Sulfamide) (Anhydrides)

Kretov, A. Ye.

Preparation of azomethine derivatives of arenequillon:
 authors: A. E. Kretov and E. A. Abrazhanova (Chem.
 Technol. Inst., Dnepropetrovsk). Zhur. Obshch. Khim.
 27, 1953-7 (1957). AlCl_3 (0.1 mole) in 150 ml. C_6H_6 treated
 dropwise with 0.1 mole chloramine-H in 50 ml. C_6H_6 at 25-
 30°, then with 0.2 mole BaH_2 , heated 5 hrs. on a steam bath,
 10 ml. 100% HCO_2H added, and the mixt. kept 1 hr., yielded
 $\text{PhSO}_2\text{N}:\text{CHPh}$ (I), m. 63-2°. I gradually decomp. on
 standing in air yielding PhSO_2NH_2 . Heating I with H_2O 2
 hrs. and allowing the mixt. to stand 1 day gave 62.5%
 PhSO_2NH_2 . A similar reaction with $\alpha\text{-O}:\text{NC}_6\text{H}_4\text{CHO}$ gave
 58.2% $m\text{-O}:\text{NC}_6\text{H}_4\text{CH}:\text{NSO}_2\text{Ph}$, m. 119-22°, while $p\text{-Me}:\text{NC}_6\text{H}_4\text{CHO}$
 gave 52.5% orange $p\text{-Me}:\text{NC}_6\text{H}_4\text{CH}:\text{NSO}_2\text{Ph}$,
 m. 192-5°. AlCl_3 (0.025 mole) in 50 ml. C_6H_6 slowly
 treated with 3.9 g. PhSO_2NH_2 , then with 2.7 g. BaH_2 and
 heated 5 hrs., yielded 44% I. Similarly, from the appropriate
 $\text{RC}_6\text{H}_4\text{SO}_2\text{NH}_2$ and $\text{RC}_6\text{H}_4\text{CHO}$ were prepd. the
 following $\text{RC}_6\text{H}_4\text{SO}_2\text{N}:\text{CHC}_6\text{H}_4\text{R}$ (R, R', % yield, and m.p.
 given): $p\text{-Cl}$, H, 63.5, 101-6°; H, $m\text{-O}_2\text{N}$, 61.2, 122-4°;
 $p\text{-Cl}$, $m\text{-O}_2\text{N}$, 150-66°; H, $p\text{-Me}:\text{N}$, 60.5, 101-5°;
 $p\text{-Cl}$, $p\text{-Me}:\text{N}$, 170-2°. Furfural gave 51.5% $\text{PhSO}_2\text{N}:\text{CHC}_6\text{H}_4\text{O}$,
 m. 126-70°, and $p\text{-ClC}_6\text{H}_4\text{SO}_2\text{N}:\text{CHC}_6\text{H}_4\text{O}$, m.
 101-2°. The probable reaction mechanisms are discussed.
 G. M. K.

4
 4E4j
 4E2c (3)
 2 May

Distr: 4E4j/4E2c(j)/4E3d

7
Reaction of 9,10-dibromoanthracene with aryl sulfon-
amides. A. R. Krasov and M. S. Rabinovitch (Chem. Tech-
nol. Inst., Dnepropetrovsk). *Zhur. Obshch. Khim.*, 27,
2774 (1957). To soln. of 1.2 g. Na in 150 ml. PhCH_2OH
was added 7.5 g. PhSO_2NH_2 and 5.4 g. 9,10-dibromoanthra-
cene, the mixt. refluxed 9 hrs., filtered, and concd., yielding
5.9 g. $\text{PhSO}_2\text{NHCH}_2\text{Ph}$, m. 83-4°. $p\text{-MeC}_6\text{H}_4\text{SO}_2\text{NH}_2$
similarly gave $p\text{-MeC}_6\text{H}_4\text{SO}_2\text{NHCH}_2\text{Ph}$, m. 113-11°, while
 $2\text{-C}_6\text{H}_5\text{SO}_2\text{NH}_2$ gave $2\text{-C}_6\text{H}_5\text{SO}_2\text{NHCH}_2\text{Ph}$, m. 130-1°. The
yields were 75-85%. The distillate during the concn.
contained aldehydes, and in case of the last reaction there
was actually isolated 90% H_2O . It is suggested that the
reaction proceeds through 9,10-bis(arylsulfonamido) deriv. of
anthracene.

G. M. Kovalevskii

my

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2 may
3

KRETOV, A.Ye.; ABRAZHANOVA, Ye.A.

Preparation of azomethine derivatives of aryl sulfamides. Zhur.ob.
khim. 27 no.7:1993-1997 J1 '57. (MIRA 10:10)

1.Dnepropetrovskiy khimiko-tehnologicheskii institut.
(Sulfamide) (Schiff bases)

KRETOV, A.Ye.; STARIKOVA, A.I.

Acylation of arylsulfamides and *N,N*-dichlorobenzene sulfamide with
phthalic acid derivatives. Ukr. khim. zhur. 24 no.3:344-347 '58.
(MIRA 11:9)

1.Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Phthalic acid) (Sulfamide)

KRETOV, A.Ye.; ROMAZANOVICH, N.P.

Chemical reactions of N,N-dicyanethylated arensulfamides. Ukr.khim.zhur.
24 no.6:761-763 '58. (MIRA 12:3)

1. Dnepropetrovskiy khimiko-tekhnologicheskoy institut im. F.M. Dzerzhin-
skogo, kafedra organicheskoy khimii.
(Sulfamide)

AUTHORS: Kretov, A. Ye., Romanovich, M. P. 79 28-4-46/60

TITLE: Cyanethylation of Arylsulfonamides (Tsianetilirovaniye arensul'fonamidov)

PERIODICAL: Zhurnal Obshchey Khimii, 1958 Vol. 28, Nr 4 pp. 1059-1062 (USSR)

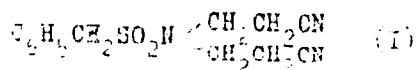
ABSTRACT: For the purpose of investigating the chemical properties of benzenesulfonamides and of their derivatives cyanethylation reactions became very important in recent time. These reactions depend on the influence of acrylonitrile upon compounds with a mobile hydrogen atom. A number of summarizing articles on cyanethylation exist (Ref 2). Acrylonitrile can react with hydrogen halides, amines, compounds of malonic acid type etc. Articles on the cyanethylation of carboxylic acids and of their derivatives (compound esters, nitriles, amides) (refs 3-5) exist as well. The synthetic peculiarities of this reaction were investigated not long ago, however, they distinctly show already now the possibility of practical application. In publications there are references to that in the hydrolysis of the products of

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Cyanethylation of Arylsulfonamides

79-28-4-46/42

cyanethylation of amine compounds are obtained which are physiologically effective (refs 6-7). As is well known in the most effective sulfonamide preparations the hydrogen atoms of the amide group are substituted (ref 8); accordingly the authors investigated the cyanethylation of benzenesulfonamide and of its derivatives. In the presence of alkalis benzenesulfonamide forms at 100°C with acrylonitrile a dicyanethyl compound (refs 9-10). Of the substituted benzenesulfonamides the benzylsulfonamide was dicyanethylated in publications. Contrary to a former assumption (ref 5) this reaction affected the amide group and the compound (I) was formed



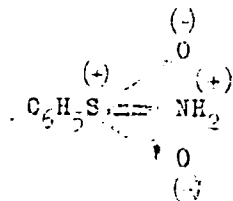
In the cyanethylation of benzenesulfonamides the conjugated position of the benzene ring to the sulfo- and amine groups are to be considered. The electrophile effect of sulfur and the separation of the free electrons causes a positive charge

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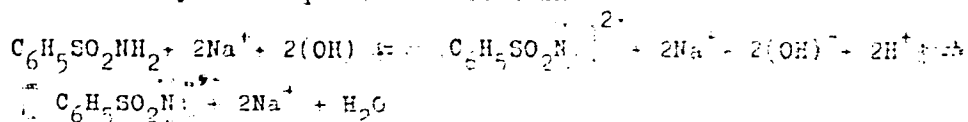
Cyanethylation of Arylsulfonamides

75-20-4 40/60

of nitrogen which facilitates the fission of a proton



This fission is still intensified in alkaline solution because a dynamic equilibrium sets in



At a molar ratio of the benzenesulfonamide to sodium hydroxide of 1:2 the anion $\text{C}_6\text{H}_5\text{SO}_2\text{N}^{2-}$ (ref 1) forms under fission of two protons. By this means the catalytic effect of sodium hydroxide in the cyanethylation of the benzenesulfonamide and the difficulty of the isolation of products of the reno

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Cyanethylation of Arylsulfonamides

10-10-140/50

cyanethylation as well are explained. At a molar ratio of 1:3 also the monovalent anion $C_6H_5SO_2NH_2$ forms under the given conditions. The carrying out of the cyanethylation of the benzenesulfonamide is exactly described. Of the derivatives of benzenesulfonamide with variously substituted benzene rings the following compounds were cyanethyated: p-toluenesulfonamide, p-chlorobenzenesulfonamide, p-methylbenzenesulfonamide, p-phenylbenzenesulfonamide and sulfidone. The compounds reproduced by this reaction have hitherto not been described in publications. The products of the cyanethylation of a number of aromatic chlorine compounds, which are used as insecticides, are described in publications. In this respect the dicyanethylation of 1-chloro-2-nitro-4-benzenesulfonamide deserves special interest. It proceeds very violently in the presence of the catalyst according to Rodionov and supplies a dicyanethyated product. The investigation of the reaction mechanism of the reactions carried out showed that the substitution of hydrogen by the cyanethyl group takes place under the described conditions only in the amide group. No reaction in the amino

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Cyanethylation of Arylsulfonamides

79-28-4-46/62

group takes place.

There are 1 table and 22 references, 7 of which are Soviet

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
(Dnepropetrovsk Chemical-Technological Institute)

SUBMITTED: March 26, 1957

Card 5/5

AUTHORS: Kretov, A. Ye., Kremlev, M. M. SOV/79-28-7-49/64

TITLE: The Reaction of the N,N-Dichlorobenzene Sulfamide With Polyhalogen Derivatives of Methane.I.(Reaktsiya N,N-dikhlorobenzol-sul'famida s poligalogenproizvodnymi metana.I)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 7, pp. 1950 - 1954 (USSR)

ABSTRACT: In the case of a heating of N,N-dichlorobenzene sulfamide (further on called dichloramine B,!) with excess carbon tetrachloride in the presence of $AlCl_3$ the authors found a considerable formation of chlorine and the formation of benzene sulfochloride, which fact points to the participation of CCl_4 . The experiments with different amounts of dichloramine and $AlCl_3$ showed the following results (Table 1): In the reaction of 3 and 4 moles of dichloramine B with one mole of $AlCl_3$ in CCl_4 even after 30 hours heating dichloramine B remains which did not enter reaction. Therefore the authors contented themselves with only two ratios between dichloramine and $AlCl_3$, viz. 2:1 and 1:1. It

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The Reaction of the N,N-Dichlorobenzene Sulfamide With SOV/79-28-7-49/64
Polyhalogen Derivatives of Methane.I.

had been shown already earlier that the anhydrous $AlCl_3$ reacts energetically with dichloramine at a ratio of 1:1 amidst aromatic hydrocarbons under the formation of an oily complex and an equimolecular amount of chlorinated hydrocarbon. However, in the case of equimolecular amounts of dichloramine B and $AlCl_3$ and excess CCl_4 the dichloramine B and $AlCl_3$ dissolve completely already after half an hour's heating ($34-35^\circ$). A heavy oil accumulates on the bottom which forms chlorine on a further heating. Besides chlorine and benzene sulfochloride also cyanuric chloride separates in the reduction. In quantitative respect the process can be represented by the following reaction process:
 $3C_6H_5SO_2NCl_2 + 3CCl_4 \rightarrow 3C_6H_5SO_2Cl + 6Cl_2 + C_3N_3Cl_3$. Two more theoretical and partially experimental considerations concerning this subject follow. There are 2 tables and 7 references, 3 of which are Soviet.

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The Reaction of the N,N-Dichlorobenzene Sulfamide With SO₂/79-28-7-49/64
Polyhalogen Derivatives of Methane.I.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut (Dnepro-
petrovsk Chemical and Technical Institute)

SUBMITTED: June 6, 1957

1. Dichlorobenzene sulfamide--Chemical reactions 2. Halogen compounds -
--Chemical reactions 3. Methanes--Chemical reactions 4. Aluminum
chlorides--Chemical effects

Card 3/3

AUTHORS: Kretov, A. Ye., Kremlev, M. M. SOV/79-28-7-50/64

TITLE: The Reaction of N,N-Dichlorobenzene Sulfamide With Polyhalogen Derivatives of Methane. II. (Reaktsiya N,N-dikhlorobenzolsulfamida s poligalogenproizvodnymi metana.II)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 7, pp 1954 - 1957 (USSR)

ABSTRACT: After the first publication (Ref 1) the authors expected that the N,N-dichlorobenzene sulfamide would also react with other polyhalogen derivatives of methane in the presence of $AlCl_3$ in similar cases, In order to investigate this in detail they added chloropicrin and chloroform. The chloropicrin was taken in excess quantities as it had to be reagent and medium at the same time, the sulfamide and $AlCl_3$, however, were taken in equimolecular quantities. Right in the beginning of the reaction the mixture becomes warm and starts a considerable formation of chlorine and $ClCN$; it was, however, found that on the action of $AlCl_3$ no decomposition of chloropicrin according to the

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The Reaction of N,N-Dichlorobenzene Sulfamide With
Polyhalogen Derivatives of Methane.II.

SOV/79-28-7-50/64

reaction $\text{CCl}_3\text{NO}_2 \rightarrow \text{COCl}_2 + \text{ClNO}$ takes place, as this reaction only begins at the boiling point when chloropicrin is heated with AlCl_3 . In the case of AlCl_3 being added to chloropicrin no chlorine formed. However on a further addition of dichloroamine B (=N,N-dichlorobenzene sulfamide) an immediate formation of chlorine and ClNO_2 began. In the case of a heating to 30° the chlorine formation becomes turbulent and a heavy yellow oil is formed. It was experimentally found that in the reaction of chloropicrin with dichloroamine B the separation of ClCN by condensation in the liquid state is beyond any doubt. Benzoyl chloride in great amounts and cyanuric chloride in small amounts were the products of side reactions. According to the amounts of chlorine and cyanogen chloride found the reaction must take place in the following way:

$3\text{C}_6\text{H}_5\text{SO}_2\text{NCl}_2 + 3\text{CCl}_3\text{NO}_2 \rightarrow 3\text{C}_6\text{H}_5\text{SO}_2\text{Cl} + \text{C}_3\text{N}_3\text{Cl}_3 + 3\text{Cl}_2 + 3\text{ClNO}_2$. The reaction with chloroform, instead of dichloramine, takes a similar course. The mechanism of the reactions takes obviously place through the free radicals, which fact could be further

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The Reaction of N,N-Dichlorobenzene Sulfamide With
Polyhalogen Derivatives of Methane.II.

SOV/79-28-7-50/64

proved by other reactions. There are 2 tables and 2 references,
2 of which are Soviet.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut (Dnepro-
petrovsk Chemical and Technical Institute)

SUBMITTED: July 1, 1957

1. Dichlorobenzene sulfamide--Chemical reactions 2. Halogen
compounds--Chemical reactions 3. Methanes--Chemical reactions
4. Aluminum chlorides--Chemical effects

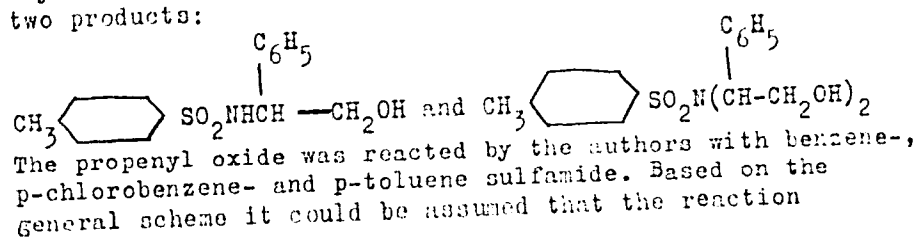
Card 3/3

AUTHORS: Kretov, A. Ye., Abrazhanova, Ye. A. SOV/79-28-10-30/60

TITLE: On the Reaction of Aryl Sulfamides With Propylene Oxide
(O reaktsii arilsul'famidov s okis'yu propilena)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 10,
pp 2779 - 2782 (USSR)

ABSTRACT: Ethylene oxide found widespread use as raw material for many products used as solvents (Ref 1), plastifiers, varnishes (Refs 2,3), in ammunition and medical preparation industries. The reactions of propylene oxide with aryl sulfamides are investigated. Jonson (Dzhonson) (Ref 5) already carried out the reaction of styrene oxide with p-toluene sulfamides, and separated two products:



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On the Reaction of Aryl Sulfamides With Propylene Oxide SOV/79-28-10-30/6.0

takes place in two steps so that the mono or di-substituted aryl sulfamides were to be expected (Reaction Scheme (7) and (2)). In the reaction under high pressure and with caustic soda as catalyst in which the authors proceeded from benzene sulfamide the N- β -hydroxy isopropyl benzene sulfamide (I) was obtained as main product. When using p-toluene- and p-chloro benzene sulfamide the reaction takes place according to the schemes (1) and (2) under the formation of substituted amides. The sirupy N- β -hydroxy isopropyl-aryl sulfamides (Formulae (A)) were purified in form of their crystalline sodium derivatives (with some molecules crystal water). The N- β , β' -dihydroxy diisopropyl-aryl sulfamides (B) are of crystalline nature. The halogenation and acylation of the hydroxyl groups was carried out to prove the structure of the products obtained. Eight aryl sulfamide derivatives not described in publications were synthesized and characterized. There are 6 references, 1 of which is Soviet.

Card 2/3

On the Reaction of Aryl Sulfamides With Propylene Oxide SOV/79-28-10-30/60

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
(Dnepropetrovsk Chemotechnological Institute)

SUBMITTED: August 2, 1957

Card 3/3

SOV/79-28-10-37/60

AUTHORS: Kretov, A. Ye., Tikhonova, G. V.

TITLE: N-Aryl Sulfamide Derivatives of Diethanol Amine (N-
arilsul'famidnyye proizvodnyye dietanolamina)

PERIODICAL: Zhurnal obshchey khimii, 1958, Vol 28, Nr 10,
pp 2808 - 2812 (USSR)

ABSTRACT: In contrast to the substituted ethanol amines, the
aryl sulfamide derivatives of the mono and diethanol
amines are almost not investigated at all. The
present paper was carried out to investigate the syn-
thesis and the properties of these compounds, first of
all of the compounds from diethanol amine and aryl
sulfochlorides
$$\text{ArSO}_2\text{Cl} + 2\text{HN}(\text{CH}_2\text{CH}_2\text{OH})_2 \rightarrow \text{ArSO}_2\text{N}(\text{CH}_2\text{CH}_2\text{OH})_2 + \text{HClHN}(\text{CH}_2\text{CH}_2\text{OH})_2$$

The final products of this reaction, the N,N-di-β-hydroxy
ethyl aryl sulfamides and their derivatives, were
used in form of their esters, just as the esters of
the N-alkyl-N-β-hydroxy ethyl aryl sulfamides as waxes
and plastifiers are used (Refs 1,2). The sulfamides
of this type hitherto obtained differ in many a respect

Card 1/3

N-Aryl Sulfamide Derivatives of Diethanol Amine

SOV/79-28-10-37/60

from those synthesized by the authors. They synthesized 10 N,N-di- β -hydroxy ethyl aryl sulfamides: $\text{ArSO}_2\text{N}(\text{CH}_2\text{CH}_2\text{OH})_2$ (see expressions for Ar in scheme 2).

The synthesis of the diethanol amine derivatives of the aryl sulfamides proceeding from equal mole amounts of diethanol amine and aryl sulfochloride in the presence of pyridine according to the well-known reaction (Refs 1,5) failed. Only the N,N-di- β -hydroxy ethyl- β -naphthalene sulfamide was in this case obtained in a yield of 25%. The rest of the compounds were synthesized according to a new method. It consisted in the reaction of the double amount of diethanol amine with the corresponding aryl sulfo chloride in the absence of bases in a 5 hours boiling in o-xylol. The N,N-di- β -hydroxy ethyl aryl sulfamides are easily obtained in pure state, depending on the solvent. Their structure was proved by the synthesis of their chlorides and bromides. The experimental part and scheme 3 give information of the halogen derivatives of the N,N-di- β -hydroxy ethyl aryl sulfamides. There are 6

Card 2/3

N-Aryl Sulfamide Derivatives of Diethanol Amine

SOV/79-28-10-37/60

references, 3 of which are Soviet.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
(Dnepropetrovsk Chemical and Technological Institute)

SUBMITTED: August 2, 1957

Card 3/3

AUTHORS: Kretov, A. Ye , Tikhonova, G. V. SOV/79-29-2-12/71

TITLE: N-Aryl Sulfamide Derivatives of Monoethanol Amine (N-Aril-sul'famidnyye proizvodnyye monoetanolamina)

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 412-415 (USSR)

ABSTRACT: The synthesis of the new N, β -oxyethylaryl sulfamides and their derivatives is not only of theoretical but also of practical importance (insecto-fungicidal activity). Contrarily to the already known sulfamide syntheses, in the work under review the authors used the method which they had earlier worked out for the synthesis of N,N-di- β -oxyethylaryl sulfamides (Ref 9):

$$\text{ArSO}_2\text{Cl} + 2\text{NH}_2\text{CH}_2\text{CH}_2\text{OH} \longrightarrow \text{ArSO}_2\text{NHCH}_2\text{CH}_2\text{OH} + \text{HCl} \cdot \text{H}_2\text{NCH}_2\text{CH}_2\text{OH}$$
 The corresponding aryl sulfochloride and monoethanol amine were heated for 5 hours on the reflux condenser in an o-xylene or benzene solution (Table 1). A part of the compounds obtained in syrup form did not crystallize. They are all easily soluble in alkalis, acetone and alcohol, more difficultly in benzene and difficultly soluble in water. For the purpose of investigating the chemical properties of N, β -oxyethylaryl sulfamides, the authors prepared sodium salts, chlorides and N-butyl or N-benzyl sub-

Card 1/2

N-Aryl Sulfamide Derivatives of Monoethanol Amine

SOV/79-29-2-12/71

stituted compounds. The synthesized N- β -oxyethylaryl sulfamide derivatives are specified in tables 2 and 3. All N, β -chloroethylaryl sulfamides are of a crystalline nature. Their solving conditions are given. All N-butyl and N-benzyl-N- β -oxyethylaryl sulfamides are transparent, syrup-like liquids. There are 3 tables and 9 references, 3 of which are Soviet.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
(Dnepropetrovsk Chemico-technological Institute)

SUBMITTED: December 30, 1957

Card 2/2

5.3610

AUTHORS: Lavrishchev, V. A., Plakidin, Val. L.,
Kretov, A. Ye.

69675
S/153/60/003/01/034/058
B011/B005

TITLE: Production of Amino Compounds by Interaction of Aromatic Halogen
Derivatives With Molten Urea

PERIODICAL: Izvestiya vysshikh uchebnykh zavedeniy. Khimiya i khimicheskaya
tekhnologiya, 1960, Vol 3, Nr 1, pp 127-129 (USSR)

TEXT: The authors state that the halogen atom in aromatic halogen derivatives during their fusion with urea is substituted by the amino group smoothly and with high yields. This applies to derivatives containing a nitro-, sulfamide-, alkylsulfonic, or arylsulfonic group in o- or p-position with respect to the halogen atom. The authors had proved previously that under these circumstances the halogen in o- and p-nitrochlorobenzene is not substituted at normal pressure, even at 250° (confirmed by Ref 4). A similar reaction proceeds with 2,4-dinitrochlorobenzene smoothly and with a high yield. The reaction with 2-chloro-5-nitrophenyl-N-methylsulfamide took place with a somewhat lower yield (69% instead of 83%). The substitution under review is not possible with halogen derivatives having only one sulfonic or sulfamide group in o- or p-position with respect to the chlorine atom. The results show that under the given circumstances the

Card 1/2

Production of Amino Compounds by Interaction of
Aromatic Halogen Derivatives With Molten Urea

69675

S/153/60/003/01/034/058
B011/B005

halogen in the ring becomes only movable under the influence of at least 2 strong electron-acceptor substituents. They may be 2 nitro-, or one nitro- and one sulfonic or sulfonamide group. The reaction does not start below 180°, and proceeds very quickly, sometimes with a vigorous generation of gas. From the mixture of reaction products, cyanuric acid is also obtained, which forms in the thermal decomposition of urea. There are 1 table and 9 references, 1 of which is Soviet.

ASSOCIATION: Dnepropetrovskiy khimiko-tehnologicheskii institut im. F. E. Dzerzhinskogo (Dnepropetrovsk Institute of Chemical Technology imeni F. E. Dzerzhinskiy) Rubezhanskiy filial nauchno-issledovatel'skogo instituta organicheskikh poluproduktov i krasiteley im. K. Ye. Voroshilova (Rubezhnoye Branch of the Scientific Research Institute of Organic Semiproductions and Dyes imeni K. Ye. Voroshilov) Kafedra organicheskoy khimii (Chair of Organic Chemistry)

SUBMITTED: March 7, 1959

Card 2/2

1.1000
1000
30/10/1966-10-1-1972

AUTHORS: Kono, Y. A. Y., Kolesnik, M. N.

TITLE: Concerning the Reaction of 9,10-Bis(chloromethyl)-anthracene With Acylhydrazonamides

PERIODICAL: Zhurnal obshchey khimii, 1966, Vol. 40, No. 11, pp. 640-649 (USSR)

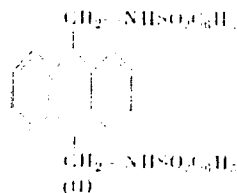
ABSTRACT: The reaction of 9,10-dihaloanthracene with acylhydrazonamides in benzyl alcohol was described previously by the authors (this j., 1966, Vol. 37, p. 2174). The present study describes the reaction of 9,10-bis(chloromethyl)-anthracene (I) with benzoylhydrazonamide in various media.



Card 1/1

Concerning the Reaction of 9,10-Bis(Chloromethyl)-anthracene With Arylsulfonamides

1955
2000-07-31-000826420



From several methods of chloromethylation the authors choose that devised by M. M. Miller, R. W. Amidon, and P. O. Tawney (J. Am. Chem. Soc., 1955, Vol. 77, p. 2845). The chloromethylation of anthracene was carried out in dioxane with paraformaldehyde and concentrated HCl, and with gaseous HCl passing through the solution. Anthracene I was obtained in 65-70% yield in the form of a powder not requiring recrystallization. Four new 9,10-bis(arylsulfonamidomethyl)-anthracenes were synthesized in reaction of I with sodium salts of arylsulfonamides in xylene or mesitylene. The sodium salts were obtained on adding a calculated amount of sodium to

Card 2/4

Concerning the Reaction of 9,10-Bis(Chloromethyl)-anthracene With Arylsulfonamides

77/11
001/19-30-2-61/78

arylsulfonamides in methanol and distilling the alcohol. The sodium salt of benzenesulfonamide thus obtained was stirred for 20 hr with o-xylene at boiling temperature. The precipitate of HCl was filtered. The filtrate, on addition of petroleum ether, yielded 9,10-bis(phenylsulfonamidomethyl)-anthracene (II; mp 112-114° C). Similarly, the reaction with p-toluenesulfonamide sodium salt gave 9,10-bis(p-toluenesulfonamidomethyl)-anthracene (mp 83-85° C), and the reactions with the corresponding sodium salts of arylsulfonamides gave 9,10-bis(p-iso-propylbenzenesulfonamidomethyl)-anthracene (mp 93-94° C) and 9,10-bis(mesitylenesulfonamidomethyl)-anthracene (mp 138-140° C). There are 7 references, 2 U.S., 2 U.K., 3 Soviet. The U.S. and U.K. references are: Stephen, Short, Glading, J. Chem. Soc., 117, 510 (1920); G. M. Badger, *ibid.*, p 802 (1939); M. W. Miller, R. W. Amidon, P. O. Tawney, J. Am. Chem. Soc., 77, 2845 (1955); E. H. Northey, Chem. Revs., 27, 85 (1940).

Card 3/4

Concerning the Reaction of 9,10-Bis-(Chloro-
methyl)-anthracene With Arylsulfonamides

7791a

207. 17-22-1-51/58

ASSOCIATION: Dnepropetrovsk Chemical Technological Institute
(Dnepropetrovskiy khimiko-tekhnologicheskiy institut)

SUBMITTED: January 29, 1959

Card 4/4

9.3620

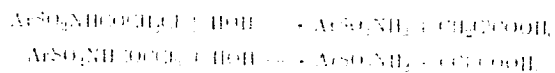
104-50-2-41/69

AUTHORS: K. H. G. Zee, K. H. G. Zee

TITLE: Hydrolysis of N-Chloro- and N-Trichloroethanesulfonamides

PERIODICAL: *Journal of Polymer Science: Polymer Chemistry Edition*, Vol. 30, No. 4, pp. 945-947 (1992)

ABSTRACT: N-Chloro- and N-trichloroacetylarylaminesulfonamides can be hydrolyzed by boiling in aqueous acidic dilute acid (5%), and alkali solution. The reaction yields arenesulfonamides, chloroacetic, and trichloroacetic acids. The latter decompose under hydrolytic conditions yielding carbon dioxide and carbon disulfide.



Cont. 1/4

Isobutylphosphoryl and isobutylidene groups hydrolysis
are mostly from H₂O and H₂O₂ respectively. The

Hydrolysis of N-ethyl- and N-Tetradecyl-
ammonium salts

67-72-30-3-41/69

Results of hydrolysis are shown in the Table. The intro-
duction of alkyls into the chain of tetradecyl- and N-
ethyl- and tetradecyl- ammonium salts, the rate of
hydrolysis. There is a linear relationship between the rate of
hydrolysis and the length of the alkyl chain.

ASSOCIATION: Department of Chemistry, University of Illinois
(Department of Chemistry, University of Illinois)

COMMITTEE: Department of Chemistry

and 2/4

Hydrolysis of the Nitro- and Sulfonamide-
 groups in Compound 10a

10a

1.	2.	3.	4.
1			
2			
3	$C_6H_5SO_2NHCOCH_2Cl$	(10)	100
4			100
5		50% H_2SO_4	100
6		50% $NaOH$	100
7	$C_6H_5SO_2NHCOCH_2Cl$	(10)	100
8			100
9	$p-CH_3C_6H_4SO_2NHCOCH_2Cl$	(11)	100
10			100
11			100
12			100
13	$2,6-(CH_3)_2C_6H_3SO_2NHCOCH_2Cl$	(12)	100
14			100
15	$C_6H_5SO_2NHCOCH_2Cl$	50% H_2SO_4	100
16		50% $NaOH$	100
17	$p-CH_3C_6H_4SO_2NHCOCH_2Cl$		100
18	$2,6-(CH_3)_2C_6H_3SO_2NHCOCH_2Cl$	(13)	100
19	$p-CH_3C_6H_4SO_2NHCOCH_2Cl$		100

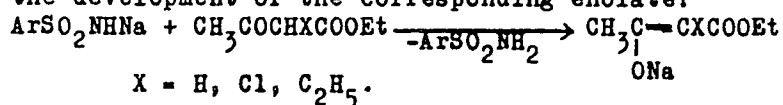
S/079/60/030/04/42/080
B001/B002

AUTHORS: Kretov, A. Ye., Abrazhanova, Ye. A.

TITLE: Acylation of Arene Sulfamides With Esters of Acetoacetic Acid
and Its Substituted Compounds

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 4, pp. 1243 - 1245

TEXT: In addition to the papers of Refs. 1,2, the authors investigated the reactions of alkali salts of arene sulfamides on the one hand, with esters of acetoacetic acid and its substituted compounds on the other. The initial substances were benzene-, p-chloro, p-toluene sulfamide and the esters of acetoacetic acid, and of chloro-, ethyl-, dichloro-, and diethylacetoacetic acid. Under the participation of a mobile hydrogen of the methylene group of the ester in the reaction, the latter proved to take place under the development of the corresponding enolate:

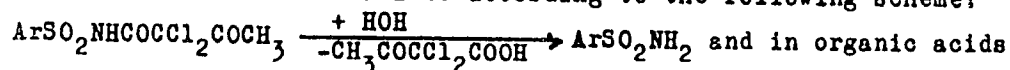


Card 1/2

Acylation of Arene Sulfamides With Esters of Acetic Acid and Its Substituted Compounds S/079/60/030/04/42/080
B001/B002

If no mobile hydrogen is present, the ester group enters into reaction:

$$\text{ArSO}_2\text{NHNa} + \text{CH}_3\text{COCl}_2\text{COOEt} \xrightarrow{-\text{EtONa}} \text{ArSO}_2\text{NHCOCCH}_2\text{COCH}_3$$
 The reaction took place in boiling, non-aqueous methanol. Due to the hydrolysis taking place readily, it was not possible to separate the final product in the pure state. The hydrolysis takes place in neutral and alkaline media, under the formation of arene sulfamides according to the following scheme:



according to a different scheme. Thus the following compounds were synthesized: N-dichloroacetylbenzene sulfamide, N-dichloroacetyl-p-toluene sulfamide, N-dichloroacetyl-p-chlorobenzene sulfamide. These are colorless, crystalline products which are soluble in hot water and benzene, and which may be titrated with phenolphthalein in an alcoholic solution at a low temperature. There are 2 references, 1 of which is Soviet.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
 (Dnepropetrovsk Institute of Chemical Technology)

SUBMITTED: April 20, 1959

Card 2/2

85391

53610 also 2209

S/079/60/030/006/019/033/XX
B001/B055

AUTHORS: Kretov, A. Ye. and Matveyev, I. S.

TITLE: V. Reaction of Cyanamide¹ With Ethylene Oxide¹

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 6, pp. 1837-1841

TEXT: Basing on three American patents (1-3) and Refs. 4-8, the authors investigated the reaction of cyanamide with excess ethylene oxide in an autoclave with mechanical stirring at comparatively low temperatures (80-65°C), with and without a solvent. Mixtures of 2-imino-oxazolidine derivatives were formed in all cases. The separation of the substances in the mixture by fractional distillation was not possible since its composition changed on heating. At 200°C in a vacuum, only 35-40% of the initial product is distilled off, the remainder gives off ammonia and turns into a solid resinous mass. The mixture was separated chromatographically on potato starch as an adsorbent and with chloroform, acetone, and methanol as solvents. The following compounds were separated and identified: 2-imino-3-β-hydroxy-ethyl-oxazolidine-1,3 (I), 2-N-β-hydroxy-ethyl-imino-3-β-hydroxy-ethyl-oxazolidine-1,3 (II), 2-N-β-hydroxy-ethyl-imino-3-
Card 1/2

85391

V. Reaction of Cyanamide With Ethylene Oxide S/079/60/030/006/019/033/XX
B001/B055

(5'-hydroxy-3'-oxa-pentyl)-oxazolidine-1,3 (III). The composition and constants are given in a table. All compounds are easily soluble in water and alcohol, moderately soluble in benzene, and insoluble in ether. Above 100°C they gradually decompose. Their structure was verified by hydrolysis of their ethers with an alkali hydroxide solution. The cyclic structure of the first-mentioned compound is demonstrated by the existence of a methoxy group in its methylation product (IV). The hydrolysis of the ether gives K_2CO_3 , methyl amine, and β -methoxy- β' -hydroxy-diethyl amine (V), thus indicating an imine structure. Hydrolysis of the dimethyl ether of the second compound (II) leads to compounds (V), (VII), and K_2CO_3 , thus also indicating an imine structure. Hydrolysis of the dimethyl ether of the third compound (VIII) yields (VII) and (IX)(imino form). There are 1 table and 8 references: 1 Soviet, 4 German, and 3 US.

ASSOCIATION: Dnepropetrovskiy khimiko-tehnologicheskii institut
(Dnepropetrovsk Institute of Chemical Technology)

SUBMITTED: June 7, 1959

Card 2/2

~~KREMONA, I. Ye.~~ SYROVATKO, A.D.

Exhaustive chlorination of methyl groups of xylenes. Zhur. ob.
khim. 30 no.9:3019-3024 S '60. (MIRA 13:9)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni F.E.
Dzerzhinskogo.

(Xylene) (Chlorination)

3/079/60/030/009/020/022/XX
B001/B066

AUTHORS: Kretov, A. Ye. and Matveyev, I. S.

TITLE: Synthesis of Amino Alcohols From Derivatives of Oxazoline
and Oxazolidine, VII.

PERIODICAL: Zhurnal obshchey khimii, 1960, Vol. 30, No. 9.
pp. 3024 - 3028

TEXT: Amino alcohols of the aliphatic and aliphatic-aromatic series, which are of considerable practical importance, can be synthesized from hardly accessible raw materials, or result as end products in low yield. Particularly complicated is the synthesis of aliphatic-aromatic amino alcohols (Ref. 3). The authors devised various methods for the synthesis of amino alcohols of both series, which are based on saponification of oxazoline and oxazolidine derivatives. The corresponding substituted oxazolines and oxazolidines are saponified with a 12% methanol solution of potassium hydroxide and heated on a boiling water bath for 45 - 60 min. Methanol is distilled from the reaction mixture,

Card 1/2

Synthesis of Amino Alcohols From Derivati- S/079/60/030/009/020/022/XX
ves of Oxazoline and Oxazolidine. VII. B001/B066

and along with it also the ammonia which is titrated with 0.1 N hydrochloric acid. The resultant potassium carbonate is separated by filtration, dissolved in water, and converted to barium carbonate by means of barium hydroxide. In pure condition the amino alcohols are obtained by fractional vacuum distillation. The primary and secondary amino alcohols synthesized are given in Table 1. The oxazoline derivatives obtained from styrene oxide and cyanamide are saponified with 50% aqueous potassium hydroxyl solution by heating for 5-6 hours on a sand bath. The mixture of the amines forms a viscous matter floating on the liquid. The aqueous solution is decanted, and the carbonate is precipitated with barium hydroxide as barium carbonate. The resultant amines are separated by chromatography (with silica gel). Benzene, chloroform, and acetone served as solvents. Silica gel and the mixture to be separated are taken in a ratio of 1 : 15. The nitrogen content of the primary amino alcohols is determined by Kjeldahl's method. There are 2 tables and 3 references.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
(Dnepropetrovsk Institute of Chemical Technology)

SUBMITTED: September 11, 1959
Card 2/2

KRETOV, A.Ye.; LITVINOV, V.V.

Chlorination of anthracene by *N,N*-dichlorophenylsulfamide. Zhur.
ob. khim. 30 no.9:3028-3031 S '60. (MIRA 13:9)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Anthracene) (Sulfamide)

LAVRISHCHEV, V.A.; PLAKIDIN, Val.I.; KRETOV, A.Ye.

Interaction of alkoxy and aryloxy derivatives of the aromatic series
with fused urea. Zhur. ob. khim. 30 no.9:3064-3072 S '60.
(MIRA 13:9)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut i Rubezhanskiy
filial Nauchno-issledovatel'skogo instituta organicheskikh
poluproduktov i krasiteley.
(Urea)

1. Author

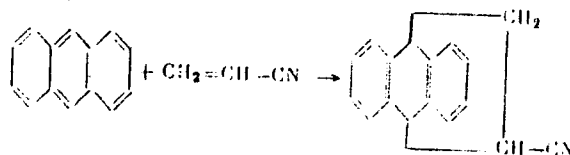
1960
744/746-33-3-44/47

AUTHORS: Rostov, A. Ye., Orlovskiy, A. Ye.

TITLE: Brief Communications. Cyanoethylation of Polycyclic Hydrocarbons

PERIODICAL: Zhurnal prikladnoy khimii, 1960, Vol 33, Nr 3, pp 744-746 (USSR)

ABSTRACT: A simple method of cyanoethylation is given for obtaining an anthracene adduct in high yield, and for its conversion into acid, amide, and esters.



Acrylonitrile was added in small portions during a 2-hr period to a boiling mixture of anthracene and acetic anhydride; boiling was continued for 22 hr.

Card 1/3

Brief Communications. Cyanoethylation of
Polycyclic Hydrocarbons

78243
SOV/80-33-3-44/47

The excess acrylonitrile and acetic anhydride was distilled under vacuum, the residue poured into water at 80-90° C, and cooled to room temperature. The oil accumulated on the surface crystallized into a hard mass which was dried, pulverized, and recrystallized from methanol. Yield of the adduct after one crystallization was 80-85%, mp 113-115° C; after triple crystallization, mp 122° C. The adduct was converted into the corresponding acid by boiling it for 5-6 hr in a saturated solution of KOH in 2-methyl-butanol, distilling the alcohol, dissolving the residue in a small amount of water, filtering, and precipitating the acid with concentrated HCl (yield 80%; mp 186° C). The amide of the adduct was obtained on heating a mixture of the latter, 12% hydrogen peroxide, 95% ethanol, and aqueous NaOH solution (yield 78-80%; mp 238.5° C). Methyl ester of the adduct was obtained on heating the latter in methanol and concentrated sulfuric acid (yield 60%; mp 115-116° C, from methanol). Butyl

Card 2/3

Brief Communications. Cyanooxylation of
Polycyclic Hydrocarbons

78243

SOV/60-33-3-44/47

ester was obtained similarly with n-butanol (yield 70-75%; mp 58-59° C). There is 1 table; and 7 references, 2 U.S., 2 German, 1 French, 2 Japanese. The U.S. references are: U.S. Pat. 1650950 (1927); U.S. Pat. 1620807 (1927).

ASSOCIATION: Dnepropetrovsk Chemical Technological Institute
(Dnepropetrovskiy khimiko-tekhnologicheskii institut)

SUBMITTED: November 12, 1959

Card 3/3

S/080/60/033/04/37/045

AUTHORS: Plakidin, Vl.L., Kretov, A.Ye.

TITLE: The Production of Acenaphthenequinone

PERIODICAL: Zhurnal prikladnoy khimii, 1960, Vol 33, Nr 4, pp 968 - 971

TEXT: A new method was developed for the production of acenaphthenequinone based on the formation of acenaphthenequinonemonooxime from acenaphthene and sodium nitrite in the presence of aqueous hydrochloric acid in a triethyleneglycol solution. The reaction is started by nitosyl chloride which is formed by the interaction of nitrous acid with hydrogen chloride. The second stage of the reaction consists in the saponification of acenaphthenequinonemonooxime by a solution of sulfuric acid in the presence of formalin. After the end of the saponification commercial acenaphthenequinone is obtained and then purified through a bisulfite compound which is formed during heating with 10 - 11%-solution of sodium bisulfite at a temperature of 100 - 102°C. The bisulfite compound is decomposed by a soda solution and the purified acenaphthenequinone is obtained with a melting point not lower than 254°C. The yield of acenaphthenequinone is 75 - 80% of the theoretical. All solvents which do not contain hydroxyl groups can

Card 1/2

The Production of Acenaphthenequinone

S/080/60/033/04/37/045

not be used for the synthesis of acenaphthenequinonemonooxime. Among the hydroxyl-containing solvents the best results were obtained with triethyleneglycol. Metal filings of copper, aluminum, iron, and stainless steel reduce the yield of acenaphthenequinone considerably.

There are: 2 tables and 6 references, 3 of which are Soviet and 3 German.

SUBMITTED: July 6, 1959

Card 2/2

OKHRAMOVICH, A.Ye.; KRETOV, A.Ye.

Synthesis and study of fluorene derivatives. Zhur. prikl. khim.
33 no.9:2148-2151 S '60. (MIRA 13:10)
(Fluorene)

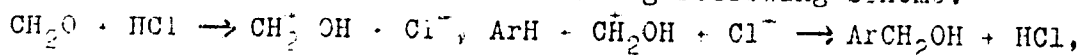
S/080/60/033/010/022/029
D216/D306

AUTHORS: Kretov, A.Ye., Silin, N.F., Korchagina, A.M.,
Lokshin, G.B., and Kitaina, S.N.

TITLE: The synthesis of terephthalic acid by chloromethylation of the products of aromatic hydrocarbons

PERIODICAL: Zhurnal prikladnoy khimii, v. 33, no. 10, 1960,
2329 - 2335

TEXT: The authors studied the synthesis of terephthalic acid from toluene and its homologues by chloromethylation. This chloromethylation is widely used in organic synthesis, being a typical electrophillic substitution reaction along following scheme:



The authors, by increasing the temperature of the reaction by 20°C, (to 70-75°C) achieved the cut in synthesis time to 12 hours while

Card 1/4

The synthesis of ...

S/080/60/033/010/022/029
D216/D306

still retaining the yields of I. Nazarov and A. Semenovskiy (Ref. 21: DAN SSSR, 12, 1437, 1956). The increase in yield of isomeric xylochlorides was obtained by changing the proportions of toluene and formaldehyde. The optimum yield of 82.5 % was obtained with the formaldehyde content of 95 % of toluene giving a molar proportion of toluene and formaldehyde of 2:1 (formaldehyde was used in form of 40 % formalin). On the chloromethylation of ethyl benzene at 70-75°C for 25 hours a maximum yield of ethyl benzyl chloride of 90 % (on ethyl benzene used) was obtained with a proportion 1:1 of ethyl benzene-formaldehyde. The optimum yield of iso-propylbenzyl chloride was 80 % on the cumene used and with a proportion of cumene:formaldehyde of 3:1, temperature 70-75°C, time 25 hours. The authors studied the oxidation of isomeric xylochlorides with dilute (10 %) nitric acid with an optimum yield of toluic acids, of 89 % for periods of 17-18 hours. Later, in connection with the discovery of nitroproducts, the concentration of acid was cut down to 7-5 % and the times to 12-10 hours. The yield obtained was 85 %. On oxidation of iso-propyl benzyl chloride, besides iso-propyl benzoic acid, whose yield was up to 80 %, 20 % of a product was obtained which

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was insoluble in a soda solution and which seemed to be a tertiary alcohol. The fractional precipitation of toluic acids was also used as a means of separation, by removing HCl from the solutions of sodium salts. *n*-toluic acid was obtained with a yield of 42.3 % and melting point 170 - 178°C, *o*-toluic acid with a yield of 40 % and a melting point 95 - 99°C. Dicarboxylic acids were also obtained with high melting points and a yield of 15.5 %. Technical literature gives various methods of esterification of terephthalic acid, but the authors obtained dimethyl terephthalate by esterification of the acid with a large excess of methanol (48 mls. to 4 g. of acid) and in the presence of concentrated sulphuric acid. This product proved unsuitable for transesterification. Esterification of dicarboxylic acids in the presence of hydrogen chloride yielded 96 % of dimethylterephthalate which did not darken on heating to 250°C. Further purification was achieved by double distillation under CO₂. The product obtained gave a melting point of 141°C, which agrees with the required standard. There are 4 tables, 1 figure and 32 references: 6 Soviet-bloc and 26 non-Soviet-bloc.

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The 4 most recent references to the English-language publications read as follows: Chem. Trade J., 143, 3717, 504, 1958; J. Bengstrom, J. Org. Chem., 23, 242, 1958; Khasimoto, Ono Khagakhamma, Annesi, J. Chem. Soc. Japan (Ind.) 59, 1136, 1956; Am. pat 2766280 1956.

SUBMITTED: March 16, 1960

Card 4/4

KRETOV, A. Ye.; MOMSENKO, A. P.

Reactions of cyanamide with organic acids. Izv. vys. ucheb. zav.;
khim. i khim. tekhn. 4, no. 1: 84-86, 1961. (MIRA 14:6)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut, kafedra
organicheskoy khimii.

(Cyanamide) (Acids, Organic)

KRETCOV, A.Ye.; MATVEYEV, I.S.

Reaction of cyanamide with propylene oxide in an aqueous medium.

Izv.vys.ucheb.zav.;khim.i khim.tekh. 4 no.3:423-425 '61.

(MIRA 14:10)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni

F.M. Dzerzhinskogo, kafedra organicheskoy khimii.

(Cyanamide)

(Propylene oxide)

S/081/62/000/021/026/069
B117/B101

AUTHORS: Matveyev, I. S., Kretov, A. Ye.

TITLE: Synthesis of nitrogenous compounds from propylene oxide and cyanamide and their reactions. 3

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 21, 1962, 187 - 188, abstract 21Zh190 (Nauchn. tr. Dnepropetr. khim.-tekhnol. in-t. no. 12, part 2, 1961, 11 - 17)

TEXT: Propylene oxide was proved to react with NH_2CN according to Krasuskiy's rule by forming a mixture of 2-imino-1,3-oxazolidine derivatives. 0.369 mole of NH_2CN , 0.862 mole of propylene oxide, and 50 mg of $\text{Ca}(\text{OH})_2$ are kept in an autoclave for 3 hrs at 65 - 75°C, the mixture being stirred every 30 - 40 min for 3 - 5 min. Thus a mixture (A) is obtained in a yield of 93 - 97%. Using 225 ml C_6H_6 (120 - 140°C, 3 hrs, 10 - 12 atm) the yield is 90 - 92%. 11 g of A is dissolved in dioxane or acetone, $\text{NH}(\text{CN})_2$ is separated, the solvent is distilled off, and 25 ml of CHCl_3 is

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added to the residue. The solution, which is separable after 48 hrs, is boiled down to one-fourth its volume and is examined by chromatography on silica gel (column, 65 times 1 cm; ratio of silica gel to A = 7:1). The following compounds are washed out as viscous liquids: 1.1 g of 2-N- β -hydroxypropyl-3-(2',4'-dimethyl-3-oxa-6'-hydroxypentyl)-5-methyl-1,3-oxazolidine, $C_{13}H_{26}N_2O_4$ (I), n_D^{20} 1.4840, d_{20}^{20} 1.0986, using 100 ml of petroleum ether; 4.4 g of 2-N- β -hydroxypropylimino-3- β -hydroxypropyl-5-methyl-1,3-oxazolidine, $C_{10}H_{20}N_2O_3$ (II), n_D^{20} 1.4750, d_{20}^{20} 1.1340, using 100 ml of $CHCl_3$; 1.6 g of 2-imino-3- β -hydroxypropyl-5-methyl-1,3-oxazolidine, $C_7H_{14}N_2O_2$ (III), n_D^{20} 1.4889, d_{20}^{20} 1.0200, using 65 ml of dioxane; and 2-amino-2-cyanamido-3- β -hydroxypropyl-5-methyl-1,3-oxazolidine, $C_8H_{16}N_4O_2$ (IV), using CH_3OH . The portion of A insoluble in $CHCl_3$ is extracted with dioxane, and 1.7 g of III and 2.2 g of IV are separated in the column. 0.048 mole of $SOCl_2$ is added to 0.03 mole of III, (temperature $< 15^\circ C$) left standing for 12 - 15 hrs, and kept at $70^\circ C$ for 2 hrs and at $100^\circ C$ for 1 hr.

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Then ice water is added, filtering takes place, and 2-imino-3- β -chloro-propyl-5-methyl-1,3-oxazolidine hydrochloride $C_7H_{13}N_2O \cdot HCl$, is extracted with butanol. 0.025 mole of I - IV is methylated with 0.1 g of CH_3I and 0.025 mole of HgO or PbO for 5 - 6 hrs at $100^\circ C$ and dissolved in water. The product is extracted with $n-C_4H_9OH$ [compound, gross formula, boiling point in $^\circ C/mm$ Hg, n_D (temperature in $^\circ C$), and d are given]: 2- β -methoxy-propylimino-3-(2',4'-dimethyl-3'-oxa-6'-methoxypentyl)-5-methyl-1,3-oxazolidine, $C_{15}H_{30}N_2O_4$, 126 - 129/1, 1.472 (27), 1.0472; 2- β -methoxypropylimino-3- β -methoxypropyl-5-methyl-1,3-oxazolidine, $C_{12}H_{24}N_2O_3$, 78 - 84/5, 1.495 (24), 1.0383; 2-methylimino-3- β -methoxypropyl-5-methyl-1,3-oxazolidine, $C_9H_{18}N_2O_2$, -, 1.5412 (18), 1.1320; 2-amino-2-cyanamido-3-methoxypropyl-1,3-oxazolidine, $C_9H_{18}N_4O_2$ (HgI_2 is extracted from an aqueous solution by ether, and the water is distilled off), -, -, -. 0.025 mole of the substance is saponified with 40 ml of a 10% KOH solution in CH_3OH and heated for 2 hrs. Then CH_3OH is distilled off from the filtrate, and the

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Following amines are obtained: from III one obtains 1,3'-dihydroxydipropylamine, $C_6H_{15}NO_2$ (V); b.p., 125 - 128°C/mm Hg; II gives V and 2'-hydroxypropylamine, C_3H_9NO (VI); b.p., 72 - 74°C/3 mm Hg; I yields VI and 1,8-dihydroxy-2,4-dimethyl-3-oxa-6-azanonane, $C_7H_{12}NO_3$; b.p., 190 - 195°C/3 mm Hg; by saponifying II with 2.5% alcoholic KOH one obtains 1'-methoxy-1'-hydroxydipropylamine, $C_7H_{17}NO_2$, and 1'-methoxypropylamine, $C_4H_{11}NO$; b.p., 100 - 105°C/3 mm Hg, n_D^{26} 1.4891. Complexes with one $H_4Fe(CN)_6$ molecule are formed by 0.05 mole of I, II, and IV in 40 ml of 10% HCl with 0.075 mole of $K_4Fe(CN)_6$ in 90 ml of water. The complexes decompose at 230, 220, and 205°C, respectively. 0.03 mole of II kept with 0.09 mole of PCl_3 at 80°C for 1 hr forms the hydrochloride of the corresponding alkyl phosphorous dichloride, $C_{10}H_{18}Cl_4N_2O_3P_2HCl$; yield, 90 - 95%; a viscous mass. [Abstracter's note: Complete translation.]

Card 4/4

KRETOV, A.Ye.; MOMSENKO, A.P.

Reactions of cyanamide with higher aliphatic acids. Zhur. ob. khim.
31 no.1:73-75 Ja '61. (MIRA 14:1)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Cyanamide) (Acids, Fatty) (Amides)

KRETOV, A. Ye.; LITVINOV, V.V.

Chlorination of fluorene by N, N-dichlorobenzenesulfonamide.
Zhur. ob. khim. 31 no.4:1183-1185 Ap '61. (MIRA 14:4)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni
F. E. Dzerzhinskogo.
(Fluorene) (Dichloramine)

KRETOV, A.Ye.; MOMSENKO, A.P.

Reactions of cyanamide with substituted aliphatic acids. Zhur.ob.
khim. 31 no.6:2000-2003 Je '61. (MIRA 14:6)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Cyanamide) (Acids, Fatty)

KRETOV, A.Ye.; LITVINOV, V.V.

Chemistry of fluorene. Part 1: New derivatives of fluorene.
Zhur.ob.khim. 31 no.8:2585-2588 Ag '61. (MIRA 14:8)

1. Dnepropetrovskiy khimiko-tehnologicheskii institut imeni
F.E. Dzerzhinskogo.
(Fluorene)

KUMTOV, A. Ye.; KUL'CHITSKAYA, N.Ye.; MAL'NEV, A.F.

Isomerism of N-arylmaleimides. Zhur.ob.khim. 31 no.8:2588-
2594 Ag '61. (MIRA 14:8)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Maleimide)

KRETOV, A.Ye.; LITVINOV, V.V.

Chemistry of fluorene. Part 2: Acylation of fluorene with maleic
anhydride after Friedel-Crafts. Zhur.ob.khim. 31 no.9:2880-2882
S '61. (MIRA 14:9)

(Fluorene) (Maleic anhydride)

KRETOV, A.Ye.; MATVEYEV, I.S.

Reaction of propylene oxide with cyanamide. Part 2. Zhur.ob.khim.
31 no.9:2885-2889 S '61. (MIRA 14:9)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni
F.E.Dzerzhinskogo.

(Propylene oxide) (Cyanamide)

KRETOV, A.Ye.; MOMSENKO, A.P.

Mechanism of the reaction of cyanamide with monobasic acids of the aliphatic series. Zhur.ob.khim. 31 no.12:3916-3921 D '61. (MIRA 15:2)

1. Dnepropetrovskiy khimiko-tehnologicheskii institut.
(Cyanamide)
(Acids, Fatty)

KRETOV, A.Ye.; ABRAZHANOVA, Ye.A.; ZLOTCHENKO, S.I.

Production of hemichloronitroso hydrocarbons. Zhur.ob.khim. 31
no.12:4043-4044 D '61. (MIRA 15:2)

(Hydrocarbons)

(Nitroso compounds)

KRETOV, A.Ye.; BESPALYY, A.S.

Methylation of dicyandiamide. Zhur.prikl.khim. 34 no.3:621-625
Mr '61. (MIRA 14:5)
(Guanidine)

15 8160

24017

S/080/61/034/006/020/020
D247/D305

AUTHORS: Okhramovich, A.Ye., Kretov, A.Ye.

TITLE: Obtaining polyesters from 9.9-dipropionic acid
fluorene

PERIODICAL: Zhurnal prikladnoy khimii, v. 34, no. 6, 1961,
1381 - 1386

TEXT: The object was to obtain polyesters from 9.9-dipropionic acid for use in the preparation of plastics and high quality lacquers. 9.9-dipropionic acid fluorene was condensed with ethyleneglycol and with glycerine in the presence of various catalysts. In each case high molecular chain form esters were formed. Preliminary research with pentaerythrite indicated that satisfactory polyester resins are obtained only after the preliminary esterification of two hydroxyl groups of pentaerythrite by unsaturated high fatty acids. To obtain polyethylene esters from 9.9-dipropionic acid fluorene: (a) In the presence of zinc chloride, zinc acetate and

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lead monoxide: Into a round bottomed 3-necked 100 ml. flask provided with a condenser, thermometer and funnel for introducing nitrogen, was placed 10 g (0.032 mole) of 9,9-dipropionic acid fluorene, 4.2 g (0.067 mole) of ethyleneglycol and 0.1 g (1) anhydrous zinc chloride; (2) zinc acetate or (3) lead monoxide. The flask was heated on a sand bath for three hours at 140-160° and then for three hours at 200-220°. The nitrogen entering the reaction vessel had been previously cleansed of oxygen and dried. At 140-160° the contents were carefully stirred twice until a homogeneous mixture was obtained. After six hours the resin was poured into a porcelain dish and fractionated using acetone as solvent; (b) In a large excess of ethyleneglycol: The method was as above using 42 g (0.67 mole) of ethyleneglycol and 0.1 g of anhydrous zinc chloride (4). On warming to 140°, the mixture becomes transparent and distillation begins continuing for three hours, during which time the temperature reaches 210°. To remove all excess of ethyleneglycol a vacuum pump is used for 15 to 20 minutes. To obtain polyglycerine esters from 9,9-dipropionic acid fluorene in the presence of zinc

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Obtaining polyesters from ...

chloride, zinc acetate and lead monoxide: The method as for polyethylene esters using 6 g (0.065 mole) of glycerine and 0.1 g anhydrous zinc chloride or zinc acetate or lead monoxide. Heat for three hours at 180-200° and then for three hours at 210-220°. (b) In a large excess of glycerine, use 60 g (0.65 mole) of glycerine and 0.1 g anhydrous zinc chloride. Heat for three hours at 200° and then under vacuum for one hour gradually raising the temperature to 235°. Conclusions: (1) Hitherto undescribed polyesters were obtained by condensing 9.9-dipropionic acid fluorene with ethyleneglycol and glycerine in the presence of catalysts and without them; (2) Esters obtained using ethyleneglycol were superior in physical and chemical properties to those obtained with glycerine; (3) Of the ten esters synthesized the best was obtained with ethyleneglycol in the presence of anhydrous zinc chloride; (4) These esters can be used for the preparation of high quality lacquers. There are 1 table and 9 references: 3 Soviet-bloc and 6 non-Soviet-bloc. The references to the English-language publications read as follows: W. Smith, J. Soc. Ch. Ind., 20, 1075, 1901; W.H.

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24017

S/080/61/034/006/020/020
D247/D305

Obtaining polyesters from ...

Carothers, Ch. Rev., 8, 353, 1931.

ASSOCIATION: Dnepropetrovskiy khimiko-tekhnologicheskii institut
(Dnepro-Petrovsk Chemical Technological Institute)

SUBMITTED: November 9, 1960

Card 4/4

ACCESSION NR: AR3000210

S/0081/63/000/006/0662/0662

SOURCE: RZh. Khimiya, Abs. 67153

AUTHOR: Kozopelyanskiy, N. S.; Kretov, A. Ye; Shapovalov, L. D.

TITLE: Synthesis of fluorene-9, 9-dipropionic acid base alkyd resins.

CITED SOURCE: Lakokrasochn. materialy i ikh primeneniye, no. 3, 1962, 36-39

TOPIC TAGS: Synthesis, fluorene-9, alkyd resins

TRANSLATION: An alkyd resin (AR), modified with vegetable oil fatty acids, was synthesized from fluorene-9, 9-dipropionic acid (I) and pentaerythritol. The reaction was conducted at 200, 220 and 240°. It was found that I has a high specific reactivity which is of particular interest in the synthesis of AR. At these temperatures, especially at 240°, I undergoes partial decarboxylation which has an adverse effect on the color of AR. To obviate this effect, syntheses were carried out with addi-

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ACCESSION NR: AR3000210

tion of water in amounts of 8-10%. Addition of water was found to be highly effective and did not affect the duration of the synthesis. An equal result is achieved by addition of xylene in amounts of 10-20%. The authors consider that in the synthesis of this AR the fatty-acid method is preferable, since it results in a high rate of reaction, good analytical indices of the resin, and makes it possible to carry out a continuous process polyesterification. The AR produced from I are readily soluble in white spirit, solvent naphtha, aromatic hydrocarbons and acetates. Varnishes consisting of 55-60% solutions of the resin in solvent naphtha, with addition of 4-5% siccative, dried at 20° tack-free within 6-8 hours and completely within 24 hours, while at 100° complete drying occurred after 2 hours. Test results are given for varnish and enamel coatings containing 38-40% zinc white. V. Latov

DATE ACQ: 16May63 ENCL: 00

SUB CODE: 00

Card 2/2

KRETOV, A.Ye.; LITVINOV, V.V.

Preparation of esters of β -2-fluorenyl- and 2,7-dichloro- β -3-fluorenylacrylic acids. Zhur.VHKO 7 no.2:239-240 '62.
(MIRA 15:4)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Fluoreneacrylic acid)

KRETOV, A.Ye.; PANASYUK, L.V.

Reaction of p-dimethylamino- and p-diethylaminobenzaldehydes
with dioxanodiamides. Zhur. ob. khim. 32 no.1:96-97 Ja '62.
(Benzaldehyde) (Amides) (MIRA 15:2)

KRETOV, A.Ye.; MATVEYEV, I.S.

Synthesis of amino alcohols from calcium cyanamide and
propylene oxide. Part 8. Zhur.ob.khim. 32 no.2:471-473
F '62. (MIRA 15:2)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Alcohols)
(Calcium cyanamide)
(Propylene oxide)

LAVRISHCHEV, V.A.; KRETOV, A.Ye.

Reaction of asymmetric derivatives of urea during melting
with 4-chloro-3-nitrophenylalkyl(aryl)sulfones and sulfamides.
Zhur.ob.khim. 32 no.2:502-506 F '62. (MIRA 15:2)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni
F.E. Dzerzhinskogo i Rubezhanskiy filial nauchno-issledovatel'-
skogo instituta organicheskikh poluproduktov i krasiteley.

(Urea)
(Sulfone) (Sulfamide)

MATVEYEV, I.S.; KRETOV, A.Ye.

Reaction of calcium cyanamide with styrene chlorohydrin. Part
10. Zhur.ob.khim. 32 no.3:974-976 Mr '62. (MIRA 15:3)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni r.E.
Dzerzhinskogo.

(Calcium cyanamide) (Benzyl alcohol)

KRETOV, A.Ye.; LITVINOV, V.V.; DUBINA, V.L.

Chemistry of fluorene. Part 3: Acylation of 2,7-dichlorofluorene
with maleic anhydride. Zhur.ob.khim. 32 no.5:1564-1567 My '62.
(MIRA 15:5)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni
F.E.Dzerzhinskogo.
(Fluorene) (Maleic anhydride)

MATVEYEV, I.S.; KRETOV, A.Ye.

Synthesis of derivatives of 2-imino-5-methyl-4,3-oxazolidine.
Part 11. Zhur.ob.khim. 32 no.10:3320-3323 0'62. (MIRA 15:11)

1. Dnepropetrovskiy khimiko-tekhnologicheskoy institut imeni
F.E. Dzerzhinskogo.

(Oxazolidine)

KRETOV, A.Ye.; LITVINOV, V.V.

Chemistry of fluorene. Part 4: Synthesis of new 2,3,7-trisubstituted fluorenes. Zhur.ob.khim. 32 no.11:3799-3801 N '62. (MIRA 15:11)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni F.E. Dzerzhinskogo.

(Fluorene)

S/080/62/035/002/022/022
D204, D302

AUTHORS: Kretov, A. Ye. and Litvinov, V. V.

TITLE: Anthracene - 9,10- diacetic acid (ADA)

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, no. 2, 1962, 464-466

TEXT: Synthesis of ADA is described in view of the potential use of the compound in producing polyesters. The following scheme was adopted: A. Dimethyl formamide was found to be the best solvent for preparing the 9,10-anthranilene dinitrile, reducing the reaction time and consumption of KCN and giving a high yield of the product. The dinitrile was not purified before hydrolysis to the final product to avoid losses. ADA was purified by dissolving it in 10% soda solution, washing with CHCl_3 , acidification, twofold recrystallization from aqueous dioxan and drying in vacuo. The melting point was 308 - 315°C, with decomposition. Quality of the 9,10-bis (chloromethyl) anthracene was observed to have a considerable influence on the yield and purity of ADA. It was also found that a certain amount of water enhanced the rate and yield of the

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Anthracene - 9,10-diacetic ...

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D204/D302

nitrile forming reaction. Synthesis of 9,10-anthracene dicarboxylic acid, attempted by the above method, proved unsuccessful. Full experimental details are given. There are 4 references: 2 Soviet-bloc and 2 non-Soviet-bloc. The references to the English-language publications read as follows: G. Rio, C.R., 236, 496, (1953); M. Miller, R. Amidon and P. Tawney, J. Am. Chem. Soc., 77, 2845, (1955).

ASSOCIATION: Kafedra organicheskoy khimii Dnepropetrovskogo khimiko-tehnologicheskogo instituta imeni F. E. Dzerzhinskogo (Department of Organic Chemistry of the Dnepropetrovsk Institute of Chemical Technology imeni F. E. Dzerzhinskiy) ✓

SUBMITTED: December 12, 1960

Card 2/2

KRETOV, A.Ye.; SILIN, N.F.; BARANOVA, Ye.I.; LOKSHIN, G.B.

Production of terephthalic acid from commercial diethylbenzene.
Zhur.prikl.khim. 35 no.4:863-866 Ap '62. (MIRA 15:4)
(Terephthalic acid) (Benzene)

S/030/62/035/004/016/022
D202/D301

AUTHORS: Fretov, A. Ye. and Shmeleva, Zh. V.

TITLE: On preparing melamine and its derivatives from dicyandiamide in organic solvents

PERIODICAL: Zhurnal prikladnoy khimii, v. 35, no. 4, 1962, 884-887

TEXT: The authors investigated the conversion of dicyandiamide into melamine organic solvents, since literature does not give information on the product quality and the presence of secondary reactions. It was found impossible to attain the theoretical yield as the main reaction was accompanied by the formation of secondary products, with the evolution of various amounts of ammonia. Under the optimum conditions, using diethylaniline, dimethylaniline and quinoline as solvents, the yield of melamine did not exceed 56 - 71%. At the same time the amount of unreacted dicyandiamide was small and varied between 1- 5%. The main admixtures in the product were alkali insoluble melam and melom which formed in approximately equal amounts. In some products small amounts of cyanomela-

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On preparing melamine ...

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D202/D301

mine were found. When ethanolamine was used as a solvent, both the amino- and hydroxyl groups of ethanolamine took part in the reaction and the residue was a complex mixture which was difficult to use in practice. The best yield of melamine (71%) was obtained by heating dicyandiamide with benzylamine. The residue consisted mainly of monobenzylmelamine which was stated to improve the quality of resins based on melamine. There are 3 tables and 18 references: 2 Soviet-bloc and 16 non-Soviet-bloc. The 4 most recent references to the English-language publications read as follows: US Pat. 2,658,892, (1953); US Pat. 2,757,513, (1956); Brit. Pat. 599,702, (1948).

ASSOCIATION: Dnepropetrovskiy Khimiko-tekhnologicheskii Institut
(Dnepropetrovsk Institute of Chemical Technology)

SUBMITTED: March 9, 1961

Card 2/2

ROVINSKIY, M.S.; KRETOV, A.Ye.; ZLOTCHENKO, S.I.

Determination of technical thiourea by the method of amperometric titration. Zav.lab. 29 no.2:154-156 '63. (MIRA 16:5)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.
(Urea) (Conductometric analysis)

KRETOV, A. Ye.; BESPALYY, A. S.

Derivatives of benzothiazolidine. Zhur. ob. khim. 33 no.1:
213-217 '63. (MIRA 16:1)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut.

(Thiazolidine)

KRETOV, A. Ye.; LITVINOV, V. V.

Chemistry of fluorene. Part 5:2,7-dichloro- β -1-fluorenoyl-
acrylic acid. Zhur. ob. khim. 33 no.1:267-269 '63.
(MIRA 16:1)

1. Dnepropetrovskiy khimiko-tekhnologicheskii institut imeni
F. E. Dzerzhinskogo.

(Fluoreneacrylic acid)